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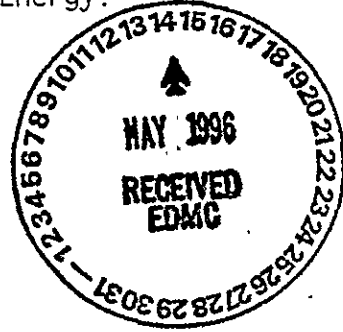
PAGE

ENGINEERING CHANGE NOTICE

Page 1 of 2


1. ECN 629979

Proj.
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Page 2 of 2

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ARCHITECT-ENGINEER

Cog. Eng. J.H. Baldwin

J.H. Baldwin

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PE

Cog. Mgr. J.G. Kristofzski

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ADDITIONAL

Tank Characterization Report for Single-Shell Tank 241-T-108

John H. Baldwin
Westinghouse Hanford Company, Richland, WA 99352
U.S. Department of Energy Contract DE-AC06-87RL10930


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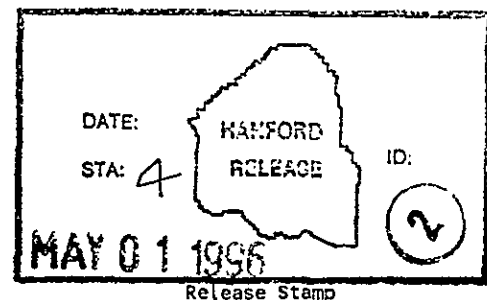
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Tank Characterization Report for Single-Shell Tank 241-T-108

J. H. Baldwin
Westinghouse Hanford Company

J. L. Stroup
ICF Kaiser Hanford Company

L. C. Amato
B. J. Morris
Los Alamos Technical Associates

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Assistant Secretary for Environmental Management



Westinghouse
Hanford Company

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Richland, Washington

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EXECUTIVE SUMMARY

This characterization report summarizes the available information on the historical uses and the current status of single-shell tank 241-T-108, and it presents the analytical results of the July 1995 sampling and analysis project. The report supports the requirements of the *Hanford Federal Facility Agreement and Consent Order*, Milestone M-44-09 (Ecology et al. 1994).

Tank 241-T-108 is the second tank in a three-tank cascade that also includes tanks 241-T-107 and 241-T-109. The tank, which entered service in September 1945, received cascade overflow from tank 241-T-107 until the first quarter of 1946 and again in the first quarter of 1953. The tank has received the following five major types of waste over its service life: bismuth phosphate first-cycle decontamination waste (1C1), tributyl phosphate waste (TBP), evaporator bottoms waste, 242-T Evaporator saltcake (T1SLTCK), and Hanford Laboratory operations waste. The Tank Layer Model (TLM) predicts that the sludge currently in the tank is composed of an upper T1SLTCK waste layer and a bottom layer of 1C1 (Agnew et al. 1995a). Although the waste contains both saltcake and sludge, the waste will be referred to as sludge to be consistent with Hanlon (1996). The tank was classified as an assumed leaker and was removed from service in April 1974. The tank was interim stabilized in November 1978, and intrusion prevention was completed in June 1981.

A description of tank 241-T-108 and its status are summarized in Table ES-1 and Figure ES-1. The tank, which has an operating capacity of 2,010 kL (530 kgal), presently contains an estimated 170 kL (44 kgal) of waste, composed entirely of sludge (Hanlon 1996).

Table ES-1. Description and Status of Tank 241-T-108.

TANK DESCRIPTION	
Type	Single-shell
Constructed	1943 to 1944
In-service	September 1945
Diameter	23 m (75 ft)
Operating depth	5.2 m (17 ft)
Capacity	2,010 kL (530 kgal)
Bottom shape	Dish
Ventilation	Passive
TANK STATUS	
Waste classification	Noncomplexed
Total waste volume	170 kL (44 kgal)
Sludge volume	170 kL (44 kgal)
Drainable interstitial liquid	0
Waste surface level (January 1991 to January 1996)	31.1 to 41.3 cm (12.25 to 16.25 in.)
Temperature (February 1976 to January 1996)	14 to 27 °C (57 to 81 °F)
Integrity	Assumed leaker
Watch List	None
SAMPLING DATES	
Auger sample	July 19 to July 21, 1995
SERVICE STATUS	
Removed from service	April 1974
Interim stabilized	November 1978
Intrusion prevention completed	June 1981

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LIST OF TERMS

1C1	first-cycle decontamination from bismuth phosphate process
ANOVA	analysis of variance
Btu/hr	British thermal units per hour
C	Celsius
Ci	curies
cm	centimeter
cm ³	cubic centimeter
DQO	data quality objective
DSC	differential scanning calorimetry
ENRAF	ENRAF-NONIUS B. V. Corporation
F	Fahrenheit
ft	feet
g	grams
GEA	gamma energy analysis
g/cm ³	grams per cubic centimeter
g/L	grams per liter
g/mL	grams per milliliter
HDW	Hanford Defined Waste
HTCE	Historical Tank Content Estimate
IC	ion chromatography
ICP	inductively coupled plasma spectroscopy
in.	inches
J/g	joules per gram
kg	kilograms
kgal	kilogallons
kL	kiloliters
kW	kilowatts
LFL	lower flammability limit
m	meter
mg	milligrams
mol/L	moles per liter
mR/hr	milliroentgens per hour
ppm	parts per million
RPD	relative percent difference
RSD	relative standard deviation
SAP	Sampling and Analysis Plan
TBP	tributyl phosphate
Temp.	temperature
TGA	thermogravimetric analysis
T1SLTCK	242-T Evaporator Saltcake
TLM	Tank Layer Model

LIST OF TERMS (Continued)

WHC	Westinghouse Hanford Company
WSTRS	Waste Status and Transaction Record Summary
wt%	weight percent
$\mu\text{Ci/g}$	microcuries per gram
$\mu\text{eq/g}$	microequivalents per gram
$\mu\text{g/g}$	micrograms per gram

2.0 HISTORICAL TANK INFORMATION

This section describes tank 241-T-108 based on recent surveillance and historical information. The first section details the present condition of the tank. This is followed by discussions of the tank's background, transfer history, and the process sources that contributed to the tank waste, including an estimate of the current contents based on the process history. Events that may be related to tank safety issues, such as potentially hazardous tank contents (ferrocyanide, organics), off-normal operating temperatures (indicative of chemical reactions), or tank damage are included. The final part of this section details the available surveillance data for the tank. Solid and liquid level data are used to determine tank integrity (leaks) and to provide clues to internal activity in the solid layers of the tank (that is, slurry growth from gas evolution with subsequent burping and collapse or shrinking caused by drying). Drywell activity monitoring is noted where anomalies may suggest leaking of the subject tank or nearby tanks. Temperature data are provided to evaluate the heat generating characteristics of the waste.

2.1 TANK STATUS

Tank 241-T-108 contains an estimated 170 kL (44 kgal) of noncomplexed waste (Hanlon 1996). Volumes of the various waste phases found in the tank are shown in Table 2-1.

Table 2-1. Summary Estimated Tank Contents Status.¹

Waste Form	Volume	
	kL	(kgal)
Total waste	170	(44)
Supernatant liquid	0	(0)
Drainable interstitial liquid	0	(0)
Drainable liquid remaining	0	(0)
Pumpable liquid remaining	0	(0)
Sludge	170	(44)
Saltcake	0	(0)

Note:

¹For definitions and calculation methods, refer to Appendix C of Hanlon (1996).

Tank 241-T-108 was classified as an assumed leaker in 1974 and removed from service in April of that year. The tank was administratively interim stabilized in November 1978; intrusion prevention was completed in June 1981. This passively ventilated tank is not on any Watch List.

2.2 TANK DESIGN AND BACKGROUND

The T Tank Farm, which was built in 1943 and 1944, is a first generation tank farm consisting of 12 tanks with a capacity of 2,010 kL (530 kgal) and four tanks with a capacity of 208 kL (55 kgal) tanks. These tanks were designed for nonboiling waste with a maximum fluid temperature of 104 °C (220 °F). Like all first generation tank farms, equipment to monitor and maintain the waste is sparse. A typical tank contains 9 to 11 risers, ranging in size from 0.1 m (4 in.) to 1.1 m (42 in.) in diameter, that provide surface level access to the underground tank. Generally, there is one riser through the center of the tank dome and four or five each on opposite sides of the tank.

Tank 241-T-108 entered service in September 1945 and is second in a three-tank cascading series. These tanks are connected by a 7.6 cm (3 in.) cascade line. The bottom center elevation of tank 241-T-107 is 193.2 m (634 ft), cascading to tank 241-T-108 at 193.0 m (633 ft), cascading to tank 241-T-109 at 192.3 m (631 ft) bottom center elevation. The height of the cascade overflow outlet is approximately 4.78 m (188 in.) from the tank bottom and 60 cm (2 ft) below the top of the steel liner. These single-shell tanks are constructed of 30 cm- (1 ft-) thick reinforced concrete with a .64 cm (0.25 in.) mild carbon steel liner (ASTM A-283 Grade C) on the bottom and sides and a 30.0 cm (1.25 ft) thick domed concrete top. These tanks have a dished bottom with a 1.2 m (4 ft) radius knuckle and a 5.2 m (17 ft) operating depth. The tanks are set on a reinforced concrete foundation. A three-ply cotton fabric waterproofing was applied over the foundation and steel tank. Four coats of primer paint were sprayed on all exposed interior tank surfaces. Tank ceiling domes were covered with three applications of magnesium zinc fluorosilicate wash. Lead flashing was used to protect the joint where the steel liner meets the concrete dome. Asbestos gaskets were used to seal the manholes in the tank dome. The tanks were waterproofed on the sides and top with tar and gunite. Each tank was covered with approximately 2.1 m (7 ft) of overburden.

The surface level is monitored through riser 13 with a manual tape (liquid level reel). In October 1995, an ENRAF gauge was installed in riser 1 to replace a defunct Food Instrument Corporation gauge. Riser 4 contains a thermocouple tree. A plan view illustrating the riser configuration is shown in Figure 2-1. A list of tank 241-T-108 risers showing the size and general use is provided in Table 2-2. This constitutes all installed equipment for tank 241-T-108.

Figure 2-2 shows a tank cross-section of the approximate waste level and a schematic of the tank equipment. Tank 241-T-108 has nine risers. Risers 2, 3, 6, and 7 (300 mm [12 in.] in diameter) and riser 5 (100 mm [4 in.] in diameter) are available. If used as sampling ports, the risers would access opposite sides of the tank.

Four tank inlets are available with one cascade inlet nozzle and one cascade overflow nozzle at approximately 4.8 m (188 in.) respectively from the tank bottom as measured at the tank wall (see Figure 2-1).

Table 2-3. Summary of Tank 241-T-108 Waste Receipt History.¹

Transfer Source	Waste Type Received	Time Period	Estimated Waste Volume	
			kL	(kgal)
T Plant/cascade from tank 241-T-107	1st cycle decontamination waste from BiPO ₄ process	1945 to 1953	4,940	(1,305)
Tank 241-TX-117	Supernate transfer from tank 241-TX-117	1954	1,707	(451)
242-T Evaporator	Evaporator bottoms saltcake from 242-T Evaporator	1955	1,934	(511)
Hanford Laboratories	Waste from laboratory operations	1967 to 1968	689	(182)
Tank 241-T-107	Supernate transfer from tank 241-T-107	1973	2,449	(647)

Note:

¹Agnew et al. (1995b) data is estimated from historical records.

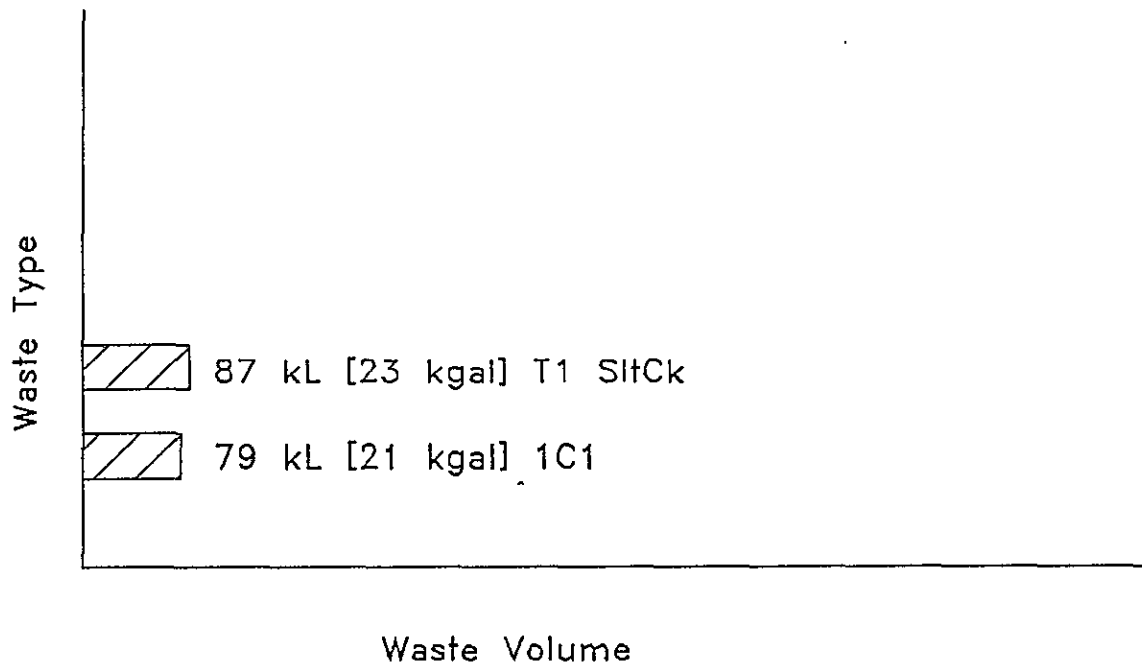
2.3.2 Historical Estimation of Tank Contents

The historical tank content estimate (Brevick et al. 1995a) is a prediction of the contents for tank 241-T-108 based on historical transfer data. However, the concentration estimates provided in the HTCE are unvalidated and should be used with caution. The historical data used for the estimate are the Waste Status and Transaction Record Summary (WSTRS) (Agnew et al. 1995b), the Hanford Defined Waste (HDW) list (Agnew 1995), and the Tank Layer Model (Agnew et al. 1995a). The WSTRS is a compilation of available waste transfer and volume status data. The HDW provides the assumed typical compositions for 50 separate wastes types. In most cases, the available data are incomplete thereby reducing the reliability of the transfer data and the modeling results derived from it. The TLM uses WSTRS data to model the waste deposition processes and HDW data to generate an estimate of the tank contents. These model predictions are considered estimates that require further evaluation using analytical data.

Based on the HTCE and the TLM, tank 241-T-108 contains a top layer of 87 kL (23 kgal) of T1SLTCK waste and a bottom layer of 79 kL (21 kgal) of 1C1 waste from the bismuth phosphate process. Figure 2-3 shows the estimated waste types and volume for the tank layers. The 1C1 layer should contain large amounts of bismuth, sodium, aluminum, nitrate, phosphate, and hydroxide. Chromium, zirconium, fluoride, iron, uranium, nitrite, silicate, and a trace of plutonium will be found as well as small quantities of strontium and cesium. Consequently, this layer will have little activity. The T1SLTCK waste should contain a very large amount of sodium. Nitrate, phosphate, fluoride, and sulfate will be present in significant quantities. Trace quantities of aluminum, iron, bismuth, chromium, uranium,

zirconium, and plutonium will be found as well. The presence of cesium and strontium will give this waste layer a correspondingly small activity, but it will be slightly larger than the 1C1 waste activity. The two waste layers are distinguished further because chloride is present in the T1SLTCK waste type but absent from the 1C1 waste and because there is a relative abundance of iron and bismuth found in 1C1 waste compared to T1SLTCK. Table 2-4 shows an estimate of the expected waste constituents and their concentrations.

Figure 2-3. Tank Layer Model for Tank 241-T-108.



2.4.1 Surface Level Readings

Because tank 241-T-108 is categorized as an assumed leaker, a manual tape is used to monitor the surface level of the waste through riser 13 daily. The leak detection criteria for tank 241-T-108 are an increase or decrease of 5 cm (2 in.) from the baseline value. The manual tape readings range from 31.1 cm (12.25 in.) to 41.3 cm (16.25 in.) from January 1991 to January 1996. A level of 33.3 cm (13.1 in.) was measured on February 12, 1996. Figure 2-4 shows a level history graph of the volume measurements.

Tank 241-T-108 does not have a liquid observation well. Six drywells are identified for this tank. Five of the six drywells exhibited large increases in radiation readings beginning around 1978. The readings peaked within the next one to three years, then slowly receded to near background levels. Initially radioactivity was thought to have originated from tank 241-T-106, but data from two new exploratory wells drilled in 1979 led to the conclusion that the activity was coming from tank 241-T-108. Erratic level readings in the years preceding the radiation increases could suggest that the tank was leaking and receiving liquid from an intrusion.

2.4.2 Internal Tank Temperatures

Tank 241-T-108 has a single thermocouple tree with 11 thermocouples to monitor the waste temperature through riser 4. Thermocouple 1 is 37.0 cm (1.2 ft) from the bottom of the tank. Thermocouples 2 through 9 are spaced at 60.0 cm (2 ft) intervals above thermocouple 1. Thermocouples 10 and 11 are at 1.2 m (4 ft) intervals.

Non-suspect data recorded between February 1976 and January 1996 from the surveillance analysis computer system were available for all thermocouples except thermocouple 1. Thermocouple 1 had data recorded between February 1976 and January 1989. Temperature data for a twelfth thermocouple were available; however, the location of this probe is unknown so the data were not considered in this report. Thermocouple 1 had a large break in data from February 1981 to July 1987. The other thermocouples had several small breaks in temperature data. The small breaks spanned nearly 33 months.

Since 1976, none of the 11 thermocouples were located within the waste. The average tank temperature above the waste was 19 °C (67 °F), the minimum was 14 °C (57 °F), and the maximum was 27 °C (81 °F). Plots of the thermocouple readings are available in Brevick et al. (1995b). Figure 2-5 shows a graph of the weekly high temperature.

water digest sample. Sampling and analytical requirements from the applicable DQOs were summarized in Table 3-1; other data for anions were obtained from the analyses as convenient (Kristofzski 1995).

Sections 3.3.1 through 3.3.6 provide a brief discussion of the sample analyses. Table 3-3 summarizes the analyses performed on samples. Quality control tests and their respective limits and requirements were performed and evaluated in accordance with the sampling and analysis plan (SAP) (Baldwin 1995c). Results of the quality control tests and the implications for data quality are discussed in Section 5.1.2.

Table 3-3. Summary of Samples and Analyses.¹

Sample Number	Auger Portion	Labcore Number ²	Analyses
95-AUG-035	Whole auger	1320	TGA, specific gravity, DSC
		1321	Total alpha, GEA
		1338	ICP H ₂ O/acid digest, IC
		1402	ICP acid digest
95-AUG-037	Whole auger	1323	TGA, specific gravity, DSC
		1324	Total alpha, GEA,
		1339	ICP H ₂ O/acid digest, IC
		1403	ICP acid digest
Flammable gas		Not applicable	LFL combustible gas meter

Notes:

¹Baldwin (1995b)

²Labcore sample numbers were abbreviated for simplification. Labcore sample numbers for auger samples 95-AUG-035 and 95-AUG-037 all contain the prefix "S95T000." Duplicate samples have the same number as the original samples.

3.3.1 Thermal Analyses - TGA and DSC

TGA and DSC analyses were performed on homogenized samples under a nitrogen purge. Sample masses ranged from 6.00 to 51.387 mg. Quality control tests included duplicates and standards.

3.3.2 Total Alpha Analysis

Total alpha activity analyses were performed on fused samples using an alpha proportional counter. Two fusions were prepared for each sample to obtain duplicate results. Quality control tests included duplicates, blanks, standards, and spikes.

3.3.3 Specific Gravity

Specific gravity measurements were performed in accordance with the requirements of the historical DQO. Quality control tests included duplicate analyses and standards. Insufficient sample precluded the duplicate analysis of sample 95-AUG-037.

3.3.4 Gamma Energy Analysis

Gamma energy analyses were performed on samples which had been prepared by a potassium hydroxide fusion procedure. Quality control tests included standards, blanks, duplicate samples, and spike recoveries.

3.3.5 Inductively Coupled Plasma Spectroscopy

Inductively coupled plasma spectroscopy analyses were performed on the acid digested waste samples to satisfy the historical DQO requirements. The Historical Program also requested ICP analysis on water digested samples. Quality control tests included standards, blanks, duplicate samples, and spike recoveries.

3.3.6 Ion Chromatography

Ion chromatography analyses were performed on water digested samples. No complexants were measured. Quality control tests included standards, blanks, duplicate samples, and spike recoveries.

Table 3-4 summarizes the analytical procedure titles, instruments, and preparation methods used to analyze tank 241-T-108 samples.

4.0 ANALYTICAL RESULTS

4.1 OVERVIEW

This section provides the analytical results associated with the auger sampling of tank 241-T-108. The sampling and analysis were performed in accordance with the SAP (Baldwin 1995c) which includes requirements for the safety screening and historical programs. The section includes a summary of the requested analytes and analytical results and a discussion of each analysis.

Table 4-1 lists the locations of the tabulated data. Although the SAP required that analyses be performed on the half-auger level, they were performed at the whole auger level because of the small size of the samples. Historical data evaluation analyses (listed as secondary analytes in Baldwin 1995c) were scheduled to be performed on the waste samples. Because of the uninteresting nature of the tank waste, however, the analyses were canceled by the Historical Program (Baldwin 1995b) except for density, ICP and GEA. In addition to the analyses required by the SAP, analyses were performed on an opportunistic basis for selected analytes in accordance with Kristofzski (1995).

Table 4-1. Analytical Data Tables.

Table Title	Table Number
Auger Sample Data Summary	Table 4-2
Thermogravimetric Analysis Results	Table 4-3
Differential Scanning Calorimetry	Table 4-4
1995 Analytical Data	Appendix A
Flammable Gas	Table 4-5

4.2 CHEMICAL DATA SUMMARY

An overall mean was calculated for each analyte by averaging concentration values for the auger samples obtained from two different risers. The results for the sample and duplicate were averaged yielding an auger mean. The two auger means were averaged to obtain an overall tank mean. This was done to assure that each auger was weighted equally. Individual sample results and their respective duplicate results are reported in Appendix A. Only a mean value and a relative standard deviation (RSD) of the mean reported in percent (defined as the standard deviation divided by the mean multiplied by 100) are reported in this section. The RSDs (mean) were calculated using standard analysis of variance (ANOVA) statistical techniques.

In addition to the overall mean, a projected tank inventory was calculated for all analytes except for energetics and percent water. The projected inventory is the product of the concentration of the analyte, the amount of waste in the tank (170 kL), and the specific gravity of 2.35. Table 4-2 summarizes the mean concentrations, relative standard deviations of the mean concentrations, and the projected inventories. Only the inventory projections from the ICP results using the acid digestions are provided in Table 4-2; the water leach results are in Appendix A.

Table 4-2. Auger Sample Data Summary.¹ (2 sheets)

Analyte	Mean	RSD (Mean)	Projected Inventory ²
Metals	($\mu\text{g/g}$)	(%)	(kg)
Al	2,290	88.0	915
Sb	< 159	n/a	< 63.5
As	< 39.8	n/a	< 15.9
Ba	< 39.8	n/a	< 15.9
Be	< 3.98	n/a	< 1.59
Bi	605	84.0	242
B	193	80.9	77.1
Cd	< 7.96	n/a	< 3.18
Ca	177	50.7	70.7
Ce	< 79.6	n/a	< 31.8
Cr	19.2	69.1	7.67
Co	< 15.9	n/a	< 6.35
Cu	< 7.96	n/a	< 3.18
Fe	6,110	89.3	2,440
La	< 39.8	n/a	< 15.9
Pb	533	81.9	213
Li	< 7.96	n/a	< 3.18
Mg	< 79.6	n/a	< 31.8
Mn	182	51.0	72.7
Mo	< 39.8	n/a	< 15.9
Nd	< 79.6	n/a	< 31.8
Ni	< 15.9	n/a	< 6.35
P	37,400	88.7	14,900
K	< 239	n/a	< 95.5

Table 4-2. Auger Sample Data Summary.¹ (2 sheets)

Analyte	Mean	RSD (Mean)	Projected Inventory ²
Metals	($\mu\text{g/g}$)	(%)	kg
Sm	< 79.6	n/a	< 31.8
Se	< 79.6	n/a	< 31.8
Si	1,500	93.0	599
Ag	< 7.96	n/a	< 3.18
Na	2.23E+05	10.2	89,100
Sr	21.6	72.4	8.63
S	371	80.0	148
Ti	< 7.96	n/a	< 3.18
Tl	< 159	n/a	< 63.5
U	1,130	79.3	451
V	< 39.8	n/a	< 15.9
Zn	52.6	52.2	21.0
Zr	10.9	45.4	4.35
Anions	($\mu\text{g/g}$)	(%)	(kg)
Br ⁻	< 6,900	n/a	< 2,760
Cl ⁻	< 905	n/a	< 362
F ⁻	10,700	88.7	4,270
NO ₃ ⁻	3.92E+05	73.9	1.57E+05
NO ₂ ⁻	6,210	73.8	2,480
PO ₄ ³⁻	1.25E+05	79.6	49,900
SO ₄ ²⁻	7,430	80.0	2,970
Radionuclides	($\mu\text{Ci/g}$)	(%)	(Ci)
Total alpha	0.0702	35.2	28.0
²⁴¹ Am	< 0.123	n/a	< 49.1
⁶⁰ Co	< 0.0133	n/a	< 5.31
¹³⁷ Cs	2.00	69.0	799
¹⁵⁴ Eu	< 0.0455	n/a	< 18.2
¹⁵⁵ Eu	< 0.0407	n/a	< 16.3

Notes:

¹Baldwin (1995b)²Projected inventories for the metals were based on the acid digestion results.

4.3 PHYSICAL DATA SUMMARY

This section discusses the physical analyses performed on the auger samples. As requested by the Historical Program, specific gravity measurements were made on the samples. Thermal analyses (TGA and DSC) were performed to satisfy the safety screening DQO.

4.3.1 Specific Gravity

Specific gravity measurements were performed using procedure LA-510-116, Rev. A-0 (Baldwin 1995b). The volume of a sludge sample with a known mass was measured by a displacement method using a nonpolar liquid. Then the specific gravity was computed by dividing the mass of the sludge sample by the mass of an equal volume of water. The specific gravity results ranged from 2.64 to 1.95 with an overall average of 2.35. The individual sample and duplicate results are in Appendix, Table A-90. There was insufficient sample for a duplicate analysis on auger sample 95-AUG-037.

4.3.2 Thermal Analyses

Thermal analyses were performed on the auger samples in accordance with the safety screening DQO. The results of the TGA and DSC analyses were used jointly to determine the ability of the waste to propagate an exothermic reaction.

4.3.2.1 Thermogravimetric Analysis. In TGA, the mass of a sample is measured while its temperature is increased at a constant rate approximately 20 to 500 °C. A gas, such as nitrogen or air, is passed over the sample while it is being heated to remove any gaseous matter. Any decrease in the weight of a sample represents a loss of gaseous matter from the sample either through evaporation or through a reaction that forms gas phase products. Water content, thermal decomposition temperatures, and reaction temperatures can be obtained from the TGA scans. The TGA for the tank 241-T-108 auger samples was performed under a nitrogen purge using procedure LA-560-112, Rev. A-2 or LA-514-114, Rev. B-0.

As shown in Table 4-3, there is a large disparity among the TGA results. Sample 1320 of auger 95-AUG-035 was reanalyzed because of the large relative percent difference (RPD) between original and duplicate results. The reanalysis results were also outside RPD limits. All results were well below the safety screening limits with a mean of 1.69 weight percent water. Notifications were not required, however, because no exothermic reactions were observed during the DSC analyses. Both the sample and duplicate for 95-AUG-037 were well above the safety screening limit, with a mean of 37.3 and a 90 percent confidence lower limit of 33.1.

Table 4-3. Thermogravimetric Analysis Results for Tank 241-T-108¹.

Sample Number	Auger Number	Temp. Range	Result	Duplicate	Mean	Overall Mean		RSD (Mean)
		(°C)	% H ₂ O	% H ₂ O	% H ₂ O	% H ₂ O		%
1320 ²	95-AUG-035	35-105	4.32	0.770	2.55	1.69	19.5	105
1320 ³	95-AUG-035	20-85	0.544	1.12	0.832			
1323	95-AUG-037	35-130	35.93	38.68	37.3	37.3		

Note:

Temp. = temperature

¹Baldwin (1995a)

²TGA performed using a Mettler™ instrument.

³TGA performed using a Perkin-Elmer™ instrument.

⁴The thermogravimetric analysis temperature range is from room temperature to 500 °C. The temperature range above is the peak width for the "result."

4.3.2.2 Differential Scanning Calorimetry. In DSC, heat absorbed or emitted by a substance is measured while the substance is exposed to a linear increase in temperature. While the substance is being heated, a gas such as nitrogen is passed over the waste material to remove gases that may be released. The onset temperature for an endothermic (characterized by or causing the absorption of heat) or exothermic (characterized by or causing the release of heat) event is determined graphically. Data generated by DSC analyses also describe heats of reaction, melting points, and solid-solid transition temperatures.

DSC analyses were performed under a nitrogen atmosphere using procedure LA-514-113, Rev. B-1, and a Mettler Model 20 differential scanning calorimeter, and procedure LA-514-114, Rev. B-0, and Perkin-Elmer equipment. No exothermic reactions were observed. No problems with quality control were noted.

The DSC results are shown in Table 4-4. The sample weight, temperature at maximum enthalpy change, and the magnitude of the enthalpy change are provided for each transition. The first transition represents the endothermic reaction associated with the evaporation of free and interstitial water. The second and third transitions probably represent the energy (heat) required to remove bound water from hydrated compounds such as aluminum hydroxide or to melt salts such as sodium nitrate.

Table 4-4. Differential Scanning Calorimetry Results for Tank 241-T-108.¹

Sample Number	Auger Number	Run	Sample Weight (mg)	Transition 1		Transition 2		Transition 3	
				Peak (°C)	ΔH (J/g)	Peak (°C)	ΔH (J/g)	Peak (°C)	ΔH (J/g)
1320	95-AUG-035	1	35.278	68.8	94.1	376.3	35.2	305.3	109.5
		2	22.354	47.1	60.0	279.0	23.3	311.3	149.0
1323	95-AUG-037	1	8.260	114.78	1,103.1	254.13	5.815	308.12	37.31
		2	6.000	99.847	954.9	---	---	---	---

Note:

 ΔH = change in enthalpy¹Baldwin (1995a)

4.4 TANK HEADSPACE FLAMMABILITY

To address flammable vapor issues, the safety screening DQO requires sampling of the tank headspace. Prior to removal of the auger samples, vapor samples were obtained from the tank headspace and analyzed using a combustible gas meter. Readings were 0 percent of the lower flammability limit (WHC 1995) indicating no flammability concerns (Table 4-5).

Table 4-5. Headspace Vapor Flammability Results for Tank 241-T-108.

Sample	Result
Flammable Gas	0% LFL

5.0 INTERPRETATION OF CHARACTERIZATION RESULTS

The purpose of this chapter is to evaluate the overall quality and consistency of the available results for tank 241-T-108 and to assess and compare these results with historical information and program requirements.

5.1 ASSESSMENT OF SAMPLING AND ANALYTICAL RESULTS

This section evaluates sampling and analysis factors that may impact the use or interpretation of data. These factors are used to assess the overall quality and consistency of the data and to identify limitations in its use. Because of the lack of analyses, some consistency checks were not possible.

5.1.1 Field Observations

Sample recovery was zero for sample 95-AUG-036 and poor for augers 95-AUG-035 and 95-AUG-037 (Baldwin 1995a). Although almost 10 in. of sample was expected from 95-AUG-035, material was found only on flutes 14 to 19 (3 in.). The amount of sample recovered was less than expected from six full auger flutes as well. Fifteen inches of sample was expected from 95-AUG-037, and material was found on flutes 5 to 19 (7.5 in.). The mass of sample was much less than expected from 15 full flutes. Therefore, the representativeness of the samples with regard to the entire tank contents may be questionable.

5.1.2 Quality Control Assessment

The usual quality control assessment includes an evaluation of the appropriate blanks, duplicates, spikes, and standards performed in conjunction with chemical analyses. All of the pertinent quality control tests were conducted on the 1995 sample results and reported in Baldwin (1995b). The SAP (Baldwin 1995c) established the specific accuracy and precision criteria for three of the quality control checks. The fourth, blank contamination, has a criterion set by the laboratory of no detected blank value being larger than five percent of the analyte concentration (DOE 1995). Sample and duplicate pairs, which had one or more quality control results outside the SAP and laboratory target levels, were footnoted in Appendix A data tables.

Both spike recoveries conducted for total alpha activity were outside the target level, and reruns produced the same results (Baldwin 1995a). However, the analytical results were far below the safety screening action limit, and deviations were not substantial enough to affect the criticality evaluation. As noted, the high levels of sodium required high dilutions for the

ICP samples. In turn, the high dilutions caused poor or meaningless spike recoveries for ICP elements that had very high concentrations or were close to the detection limit. The RPDs were similarly affected for these elements.

The laboratory analytical precision is estimated by the RPD, which is defined as the absolute value of the difference between the primary and duplicate samples, divided by their mean, times one hundred. A number of duplicate pairs had RPDs larger than the SAP limits, but most or all were caused by sample heterogeneity or large sample dilutions (ICP only). The crystalline sample material did not easily lend itself to complete homogenization. Finally, no sample violated the criterion for preparation blanks; therefore, contamination was not a problem for any analysis.

In summary, the vast majority of the quality control results were within the boundaries specified in the SAP (Baldwin 1995c). As noted in Appendix A tables, some samples did have quality control results outside SAP boundaries. However, an evaluation of quality control discrepancies has been made, and these discrepancies have not been found to impact data validity or use.

5.1.3 Data Consistency Checks

Comparing different analytical methods can be beneficial in assessing data consistency and quality. Several comparisons were possible with the data set provided by the two auger samples including the comparison of phosphorus and sulfur as analyzed by ICP with phosphate and sulfate as analyzed by IC and the calculation of a mass and charge balance. Other consistency checks, such as total alpha or total beta compared to the sum of the individual alpha or beta emitters, were not possible because of the lack of data.

5.1.3.1 Comparison of Results from Different Analytical Methods. The following data consistency checks compare the results from two or more analytical methods for a given analyte. A close correlation between the two methods strengthens the credibility of both results; a poor correlation brings the reliability of the data into question.

The analytical phosphorus mean result determined by ICP (water wash) was 18,700 $\mu\text{g/g}$, which is equivalent to 57,500 $\mu\text{g/g}$ of phosphate. This compares poorly with the IC phosphate results of $1.25\text{E}+05$ $\mu\text{g/g}$, with an RPD of 73.9. The mean ICP sulfur result (water wash) was 145 $\mu\text{g/g}$, which is equivalent to 434 $\mu\text{g/g}$ of sulfate. The RPD between this result and the result of the IC sulfate analysis of 7,430 $\mu\text{g/g}$ is 178. Both the phosphate-phosphorus comparison and the sulfate-sulfur comparison should be closer because both tests measure water-soluble species.

5.1.3.2 Mass and Charge Balance. The principle objective in performing a mass and charge balance is to determine whether measurements were consistent. When calculating the balances, only the analytes listed in Table 4-2, which were detected at a concentration of 2,000 $\mu\text{g/g}$ or greater, were considered.

Except for sodium, all cations listed in Table 5-1 were assumed to be in their most common hydroxide or oxide form, and the concentrations of the assumed species were calculated stoichiometrically. Because precipitates are neutral species, all positive charge was attributed to the sodium cation. The anionic analytes listed in Table 5-2 were assumed to be present as sodium salts and were expected to balance the positive charge exhibited by the cations. Sulfur is considered to be present as the sulfate ion and phosphorus as the phosphate ion. Both species are assumed to be completely water soluble and appear only in the anion mass and charge calculations. The concentrations of the cationic species listed in Table 5-1, the anionic species listed in Table 5-2, and the percent water were used to calculate the mass balance. The uncertainty estimates (RSDs) associated with each analyte are also listed in the tables. The uncertainty for the cation and anion totals, as well as the overall uncertainty estimate given in Table 5-3, were computed by a statistical procedure known as the propagation of errors (Bennett and Bowen 1988).

The mass balance was calculated from the formula below. The factor 0.0001 is the conversion factor from $\mu\text{g/g}$ to weight percent.

$$\begin{aligned} \text{Mass balance} &= \% \text{ Water} + 0.0001 \times \{\text{total analyte concentration}\} \\ &= \% \text{ Water} + 0.0001 \times \{\text{Al(OH)}_3 + \text{FeO(OH)} + \text{Na}^+ + \text{F}^- + \text{NO}_3^- + \text{NO}_2^- + \text{PO}_4^{3-} + \text{SO}_4^{2-}\} \end{aligned}$$

The total analyte concentration calculated from the above equation was $7.80\text{E}+05 \mu\text{g/g}$. The mean weight percent water obtained from thermogravimetric analysis shown in Table 4-2 is 19.5 percent. The mass balance resulting from adding the percent water to the total analyte concentration is 97.5 percent (see Table 5-3).

The following equations demonstrate the derivation of total cations and total anions, and the charge balance is the ratio of these two values.

$$\text{Total cations (microequivalents)} = \text{Na}^+/23.0 = 9,700 \text{ microequivalents}$$

$$\text{Total anions (microequivalents)} = \text{F}^-/19.0 + \text{NO}_3^-/62.0 + \text{NO}_2^-/46.0 + \text{PO}_4^{3-}/31.7 + \text{SO}_4^{2-}/48.1 = 11,100 \text{ microequivalents}$$

The charge balance obtained by dividing the sum of the positive charge by the sum of the negative charge was 0.874.

In summary, the above calculations yield reasonable (close to 1.00 for charge balance and 100 percent for mass balance) mass and charge balance.

Table 5-1. Cation Mass and Charge Data.

Analyte	Concentration ($\mu\text{g/g}$)	Assumed Species	Concentration of Assumed Species ($\mu\text{g/g}$)	RSD (Mean) (%)	Charge ($\mu\text{eq/g}$)
Al	2,290	$\text{Al}(\text{OH})_3$	6,620	88.0	0
Fe	6,110	$\text{FeO}(\text{OH})$	9,720	89.3	0
Na	2.23E+05	Na^+	2.23E+05	10.2	9,700
Totals			2.39E+05	10.5	9,700

Table 5-2. Anion Mass and Charge Data.

Analyte	Concentration ($\mu\text{g/g}$)	RSD (Mean) (%)	Charge ($\mu\text{eq/g}$)
F^-	10,700	88.7	563
NO_3^-	3.92E+05	73.9	6,320
NO_2^-	6,210	73.8	135
PO_4^{3-}	1.25E+05	79.6	3,940
SO_4^{2-}	7,430	80.0	155
Totals	5.41E+05	57.3	11,100

Table 5-3. Mass Balance Totals.

	Concentrations ($\mu\text{g/g}$)	RSD (Mean) (%)
Total from Table 5-3	2.39E+05	10.5
Total from Table 5-4	5.41E+05	57.3
Water %	1.95E+05	105
Grand Total	9.75E+05	38.1

5.2 COMPARISON OF HISTORICAL AND ANALYTICAL RESULTS

Because of a lack of historical sampling data, no comparisons between current and historical analytical results were possible.

5.3 TANK WASTE PROFILE

One of the objectives of the 1995 sampling event was to evaluate the tank layer model by providing a 10-in. vertical profile of the waste from two widely-spaced risers (Baldwin 1995c). The second condition was met, but a vertical profile was not obtained because only the lower half of the augers retrieved sample (which was all saltcake), therefore both auger samples were homogenized and analyzed on a whole segment basis. Information on the possible vertical disposition of the waste is available only from the TLM (Agnew et al. 1995a). According to the TLM, the waste is composed of two layers. The bottom 21 kgal is predicted to be 1C1 waste; the upper layer, T1SLTCK. The compositions of the two waste types differ (see Section 2.3.2); therefore, the tank contents were expected to be vertically heterogeneous. From the extrusion observations, however, the sampled waste appeared similar. Furthermore, these observations suggest that only saltcake was sampled. Because of the close proximity of the sampling risers to the tank walls, it is probable that the waste in the tank's dished bottom was not sampled. If 21 kgal of 1C1 waste is present as predicted, it would equate to 15 in. of waste, 12 of which would comprise the dished bottom. Surveillance data provide a surface level measurement of 13.1 in. as measured from the base of the sidewall (does not include the dish). Because only 10 in. of the waste was sampled by the augers, the 3 in. above the dish were not sampled. Therefore, it is possible that the none of the 1C1 waste was sampled.

Although multiple segments were not available for a vertical analysis of the tank waste, the fact that two risers were sampled allowed a statistical procedure known as the one-way analysis of variance (ANOVA) to be conducted to determine whether there were any horizontal differences in analyte concentrations. Analyses were calculated only for analytes where half or more of the individual measurements were above the detection limit, except for ICP water-digested results. For the ICP analytes, only acid-digested results were used. The ANOVA generates a p-value which is compared with a standard significance level ($\alpha = 0.05$). If a p-value is below 0.05, there is sufficient evidence to conclude that the sample means are significantly different from each other. However, if a p-value is above 0.05, there is not sufficient evidence to conclude that the samples are significantly different from each other.

The results of the ANOVA tests indicated that 22 of 25 analytes had significant concentration differences between the two risers. Except for sulfur (p-value = 0.083), all other 16 metals were significantly different. All five anions tested were significantly different as well as percent water and ^{137}Cs , but total alpha activity (p-value = 0.145) and density (p-value = 0.889) were not significantly different. Of the 22 analytes which had significant differences between risers, only sodium, nitrate, nitrite, and sulfate had larger concentrations at riser 5

(95-AUG-035) than at riser 2 (95-AUG-037). This does not appear to be caused by the location of the overflow inlet into the tank, because this inlet is almost equidistant between the two risers. The large discrepancy between the two auger samples could be caused by sample preparation. Sample homogenization can be very difficult with crystalline solids. In addition, the large difference in water content between the augers could affect the analytical results.

In summary, the available evidence suggests horizontal heterogeneity of the waste. Vertically, the TLM predicts two layers of waste are present, but this prediction was not verified visually and could not be verified statistically.

5.4 COMPARISON OF ANALYTICAL AND TRANSFER DATA

The concentrations of various waste constituents in tank 241-T-108 are shown in Table 5-4 along with the 1995 analytical results (from Table 4-2). Comparing the HTCE with the analytical values produced moderate to poor data correlation. A total of 18 analytes were compared. Three analytes (nitrite, fluoride, and phosphate) had RPDs under 29 percent. Four analytes (silicon, iron, density, and sodium) exhibited RPDs from 39 to 57 percent. The RPDs for the remaining 11 analytes ranged from 104 to 194 percent.

Table 5-4. Comparison of Historical Tank Content Estimate Data with 1995 Analytical Results for Tank 241-T-108. (2 sheets)

Analyte	1995 Analytical Result	HTCE Data	Relative Percent Difference
METALS	$\mu\text{g/g}$	$\mu\text{g/g}$	%
Al	2,290	12,600	138
Bi	605	6,800	167
Ca	177	2,840	176
Cr	19.2	339	179
Fe	6,110	9,540	44
Si	1,500	2,230	39
Na	2.23E+05	1.24E+05	57
U	1,130	228	132
Zr	10.9	717	194
IONS	$\mu\text{g/g}$	$\mu\text{g/g}$	%
F ⁻	10,700	8,210	26
NO ₃ ⁻	3.92E+05	75,100	136
NO ₂ ⁻	6,210	4,820	25
PO ₄ ³⁻	1.25E+05	94,000	28.3
SO ₄ ²⁻	7,430	23,500	104

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